

Deformation of olivine at mantle pressure using the D-DIA

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Abstract: Knowledge of the rheological properties of mantle materials is critical in modeling the dynamics of the Earth. The high-temperature flow law of olivine defined at mantle conditions is especially important since the pressure dependence of rheology may affect our estimation of the strength of olivine in the Earth's interior. In this study, steady-state high-temperature (up to 1473 K) deformation experiments of polycrystalline olivine (average grain size $\leq 10\ \mu\text{m}$) at pressure up to 9.6 GPa, were conducted using a Deformation-DIA (D-DIA) high-pressure apparatus and synchrotron X-ray radiation. The oxygen fugacity (f_{O_2}) during the runs was in-between the iron-wustite and the Ni/NiO buffers' f_{O_2} . The water content of the polycrystalline samples was generally about 150 to 200 wt. ppm but was as low as 35 wt ppm. Typically, 30 % strain was generated during the uniaxial compression. Sample lengths during the deformation process as well as the differential stresses were monitored *in situ* by X-ray radiography and diffraction, respectively. The strain rate was derived with an accuracy of $10^{-6}\ \text{s}^{-1}$. Differential stress was measured at constant strain rate ($\sim 10^{-5}\ \text{s}^{-1}$) using a multi-element solid-state detector combined with a conical slit. Recovered specimens were investigated by optical and transmission electron microscopy (TEM). TEM shows that dislocation glide was the dominant deformation mechanism throughout the experiment. Evidence of dislocation climb and cross-slip as active mechanisms are also reported. Deformation data show little or no dependence of the dislocation creep flow with pressure, yielding to an activation volume V^* of $0 \pm 5\ \text{cm}^3/\text{mol}$. These new data are consistent with the high-temperature rheological laws at lower pressures, as reported previously.

Key-words: olivine, high pressure, high temperature, synchrotron X-ray imaging, stress, strain, TEM, dislocations, flow, activation volume.

Introduction

Olivine is the most abundant material in the upper mantle. Accurate determination of the rheological properties of olivine at high pressure and high temperature is critical to understand the dynamic processes of the upper mantle. There are still vigorous debates about the dominant flow mechanism in the upper mantle due to the lack of knowledge on the effect of pressure on the rheological properties of olivine. The main challenge is to measure the stress and strain rate at mantle pressure (P) and temperature (T) conditions of a specimen that has reached steady state. Conventional rock deformation methods can precisely apply a force or a strain rate to define the flow of olivine (Durham & Goetze, 1977; Kirby, 1978; Kohlstedt *et al.*, 1984; Karato *et al.*, 1986; Green & Borch, 1987; Paterson, 1990; Hirth & Kohlstedt, 1995; Chopra, 1997; Mei & Kohlstedt, 2000b), although at pressure significantly lower than 3 GPa while mantle pressure is in excess of 13 GPa at the transition zone boundary.

Torsion apparatus has been applied in studying the high shear strain deformation with pressure up to 0.3 GPa (Bystricky *et al.*, 2000). Griggs-type solid medium apparatus is able to accommodate pressure up to 2.5 GPa (Ross *et al.*, 1979; Green & Borch, 1987; Karato & Jung, 2003). Molten salt cell assemblies are able to reach pressures up to 2.2 GPa (Borch *et al.*, 1989; Green & Borch, 1989). Development of the large volume press (*e.g.*, Bussod *et al.*, 1993) allowed the synthesis of deformed samples at upper-mantle pressure for microstructure examination (Karato, 1989; Karato *et al.*, 1993; Rubie *et al.*, 1993; Ando *et al.*, 1997; Cordier *et al.*, 2002). More recently, the implementation of the large volume press at X-ray synchrotron beamlines (Weidner *et al.*, 1992a; Wang *et al.*, 1988) provided the possibility of investigating *in situ* the flow properties of olivine at higher pressure. However, there existed technical limitations in controlling the applied stress and strain during deformation (Weidner *et al.*, 1992b, 1994c, 1994a, b; 1996; Li *et al.*, 2003, 2004a; Raterron *et al.*, 2004). The increasing maturity

of the Deformation-DIA apparatus (hereafter referred to as the D-DIA) (Durham *et al.*, 2002; Wang *et al.*, 2003) at synchrotrons initiated a new era for rock deformation, that is, to study steady-state rock deformation at mantle P-T conditions. The high flux of the synchrotron X-ray provides a measure of the stress from the sample itself, rather than the force/unit area across the end of the cell assembly. X-ray diffraction defines the elastic strains of subsets of grains grouped by their orientation (Li *et al.*, 2004b). Here we report the first *in situ* measurements of stress and strain rate of polycrystalline olivine at mantle pressure and temperature after achieving steady-state deformation in the D-DIA.

Leading up to the current studies, deformation experiments were conducted on olivine using a multi-anvil apparatus, but with a lack of control of stress or strain rate (Li *et al.*, 2003, 2004a; Raterron *et al.*, 2004). The differential stress and strain were measured simultaneously under high pressure (~ 8 GPa) and temperatures up to 1473 K, using a solid-state multi-detector and an imaging system (Vaughan *et al.*, 2000). Dynamic recrystallization assisted dislocation creep was observed as the dominant flow mechanism for olivine at mantle conditions. We present here the experimental data of deforming olivine in steady state condition using the D-DIA. Olivine samples were deformed at high temperature and several pressures ranging from 3.5 to 9.6 GPa. Using synchrotron X-ray diffraction as the probing tool, differential stresses were measured at constant strain rate ($\sim 10^{-5}$ s $^{-1}$) as a function of temperature. Finite strain of about 30 % was achieved during the experiments. These conditions make it possible to monitor the strength of the sample as a function of strain at mantle P-T conditions. We find that the stress-strain rate follows a similar law as that reported from low pressure experiments (Chopra & Paterson, 1984; Karato & Wu, 1993; Hirth & Kohlstedt, 1996; Mei & Kohlstedt, 2000b). Our interpretations of these results imply that the low pressure data cannot be extrapolated to mantle pressures using an activation volume greater than 5 cm 3 /mol.

Sample description

The data in this paper are from three experimental runs carried out in the D-DIA at the National Synchrotron Light Source (NSLS, Upton, NY) on the old X17B1 beamline and the new X17B2 beamline. San-37, carried two samples prepared as described below. San-72 and Gar-25 contained only one olivine sample. The experiments span the range of pressures from 3.5 to 9.6 GPa and temperatures from 1073 to 1473 K.

We use powdered San Carlos olivine ($\text{Mg}_{0.9}\text{Fe}_{0.1}\text{SiO}_4$) as the starting material. Powdered samples were finely ground in an agate mortar from optically clear (gem quality) 5 to 10-mm size crystals. The resulting grains were sorted in size using an ethanol sedimentation method. Two olivine powders of different average grain size, obtained after different sedimentation times and further called fine-grain (0.5 μm) and coarse-grain (5.0 μm) powders (± 0.2 μm , for a volume fraction > 0.9), were collected and oven-dried for a minimum of 72 hours at 433 K. The experiment San-37 contained both grain-size samples placed on top of the each other and sepa-

rated by Ni foils. Runs San-72 and Gar-25 contained the 0.5 μm starting-material only.

Water content

The water content of the starting olivine single crystals was less than 1 ppm by weight, as measured using the IR facilities at the beam line U2A of the NSLS (Wu, 2000). Despite the usual precaution to prevent atmospheric water from entering the high-pressure assembly prior to the deformation experiments, significant amounts of water was detected in the recovered olivine samples. San-37, measured at U2A of NSLS contained 150 wt ppm of water. San-72 and Gar-25 were both characterized with an IR system at the Université des Sciences et Technologies de Lille, France and contained 235 and 35 wt ppm of water respectively. These values come from the IR absorption in the 2950 – 3780 cm $^{-1}$ band, a region dominated by stretching vibrations of the O-H bonds, obtained from polycrystalline run products cut and polished to a thickness of about 100 microns. We used the same calibration as Kohlstedt *et al.* (1996) for quantifying the water content. The values must be considered to be accurate to no better than $\pm 50\%$. Thus, all of the samples have a water content of about 200 wt ppm except Gar-25, for which the water content is about a factor of 7 less.

Samples that have been quenched at different points along the pressurization-heating path have also been characterized at the NLSL facility. As described in Li *et al.* (2003), two samples were recovered along the same P-T path as those followed in the present experiments, but quenched at much earlier stages in the process in order to characterize sample microstructure and water content at the beginning of the deformation experiment. One sample was compressed to about 5 GPa, heated to 1000 K and then quenched within a few minutes. The second sample was heated to 1300 K before quenching. Both samples exhibit a well equilibrated microstructure, as revealed by TEM, with clean grain boundaries and relaxed triple junctions. IR analysis of these recovered samples revealed virtually identical water content (within experimental precision) with that measured in San-37 run products, using identical starting material and cell assemblies. We thus conclude that the OH-related defects must have entered the olivine samples at the very early stage of the experiments, and that olivine water content remained constant throughout the deformation experiments.

While we are still uncertain of the origin of the water in the samples, we note that the driest sample, Gar-25 (about an order of magnitude dryer than the other samples) was contained in a high pressure cell made of mullite, while all the other samples were inside cells made of amorphous boron bound by epoxy. We thus suspect that the epoxy binder may be the contributor of the water in these experiments.

Nonetheless, the results presented here constrain the olivine flow at fixed water content of 200 ± 50 wt. ppm with some information, from the Gar-25 sample, on the effect of different water contents on the flow properties. It is important to note, as Mei & Kohlstedt (2000a) indicate, that the IR measurement on polycrystalline samples may considerably overestimate the OH-related defect concentration in the ol-

ivine bulk. Their measurements of the water content of the polycrystalline sample that was in the same experimental charge as a single crystal indicated a water enrichment in the polycrystalline sample by a factor of 15 to 20 compared to the single crystal; a result that they interpret as the presence of OH-related defects at grain boundaries and/or in fluid inclusions in the polycrystalline olivine.

200 wt ppm, a nominal value for our 'wet' samples, corresponds to the amount of water that Kohlstedt *et al.* (1996) report for single crystal olivine at about 3.5 GPa in equilibrium with free water. By 10 GPa this becomes about 1/6 of saturation. Thus, if all the water in our samples is bound in the crystal structure, our lowest pressure data is for saturated olivine, while the highest pressure data is for significantly undersaturated olivine. Conversely, if the water mostly resides at grain boundaries as with Mei & Kohlstedt (2000a) then our samples can be considered as dry. The most significant conclusion is that the water content in our olivine samples was fixed during the deformation experiments. Our 'dry' sample, Gar-25 is at less than 5 % of saturation if all of the water is structurally bound. Thus, the comparison of the flow stress of olivine for our data should reveal the effect of pressure on the flow properties of olivine with a fixed water content and provide some insights on the effect of water on the high pressure flow in olivine.

Grain size

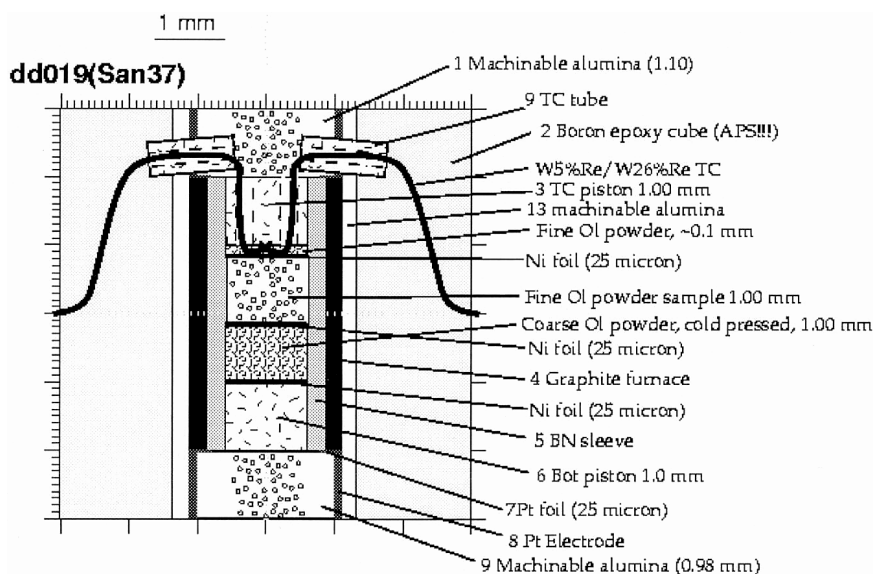
Despite the order of magnitude difference in the initial grain size of the simultaneously run samples (San-37), the grain sizes of all of the recovered samples are similar including Gar-25 and San-72. In the rheological studies, the grain growth process merges the grain size. As reported by Li *et al.* (2003), substantial grain growth occurs very early in the heating cycle. Samples that begin with the two grain sizes differing by an order of magnitude, differ by only a factor of two after heating to 1300 K and then quenched. Further heating to 1500 K for one hour reduces the grain size differ-

ence to a factor of about 1.7. We have subsequently found that stress-free heating to 1500 K can result in grain growth to sizes greater than 20 μm (from 5 μm) in about one hour. This leads us to believe that the two samples in run San-37 were of very similar grain sizes (less than a factor of two) throughout the deformation experiment and similar to those of Gar-25 and San-72 which came from the fine starting material and exhibit similar grain sizes after the run (see section on microstructures). Li *et al.* (2003) also give evidence for grain-size insensitive flow in similar conditions as we report here. We will treat the data from all samples as though they had the same grain sizes throughout the runs. Indeed, a consistent model results from this analysis. In the following text, the sample corresponding to the fine grain-size or coarse grain-size starting materials are labeled "f sample" and "c sample", respectively. This nomenclature is used to distinguish between the two samples and does not refer to the effective sample grain sizes that were very similar throughout run San-37.

Experimental protocol

The experiments reported here (run San-37, San-72 and Gar-25) were performed with the D-DIA, at NSLS old X17B1 and new X17B2 beamlines. Samples were placed in a boron nitride capsule that insulated them from a graphite furnace. The furnace, surrounded by a machinable alumina sleeve, was inserted inside an amorphous boron and epoxy pressure medium. Gar-25 used a mullite pressure medium. Differential stress was produced in the specimens with hard alumina pistons in the cell assembly (Fig. 1), placed on both ends of the specimens, that were pushed by independent rams, *i.e.* the two independent vertical anvils of the D-DIA. Samples were separated from each other (run San-37) or from the alumina pistons by Nickel foils that were used as strain markers for measuring the total strain (elastic and plastic strain) of the specimens. These Ni foils remained as pure Ni at the end of the run. This suggests that f_{O_2} was

Fig. 1. Cell assembly used in multi-anvil high-pressure deformation experiment in runs San37 and San-72 with D-DIA. Gar-25 was similar but with side entry thermocouples. The pressure medium is a boron epoxy cube with edge length 6 mm. The diameter of the inner cylindrical hole is 2.2 mm. The graphite furnace with wall thickness of 0.5 mm is contained in an alumina sleeve. The samples were contained in a boron nitride sleeve that is placed inside the furnace. A W3 %Re-W25 %Re thermocouple, situated next to the specimen, was used to measure the sample temperature. Alumina pistons were placed at the two sides of the sample to generate differential stress.



maintained during the run below the Ni/NiO-buffer oxygen fugacity, although it remained above that of the iron/wustite buffer, as revealed by the clear aspect of the run products and the absences of Ni/Fe precipitates in the recovered olivine. A W3%Re-W25%Re thermocouple was placed in one of the alumina pistons and touching one end of the samples, except for run Gar-25 where it touches the sample from the side-entry. A solid-state multi-detector (energy dispersive spectrometer, EDS) together with a conical slit was used for elastic strain measurement. In order to properly collect the X-ray beams diffracted by the samples at a 2θ angle of 6.5° , cubic boron nitride anvils (for San-37) and sintered diamond anvils (for San-72 and Gar-25), which are transparent to X-rays, were used.

Differential stresses were deduced using olivine elastic constants from specimen elastic strains which were measured as a function of time using the X-ray EDS spectra collected *in situ* in different orientations with respect to the incident beam. Following the method described by Weidner *et al.* (1992b), specimen elastic strains were measured using a white X-ray beam ($\sim 50 \times 50 \mu\text{m}^2$ in section) that passes through the polycrystalline San Carlos olivine samples in the radial direction, and probes the axial and radial lattice strains (elastic strain) of the samples while diffracted X-ray beams are recorded. With a larger incident X-ray beam ($2 \times 2 \text{ mm}^2$ in section), downstream X-ray radiographic images were captured and magnified (Fig. 2) yielding quantification of the total specimen strain.

During the experiment, the top and bottom rams as well as the main ram in the D-DIA (Durham *et al.*, 2002; Wang *et al.*, 2003) are driven at a specific speed (oil flux) to maintain both constant pressure and strain rate. The speed of the movement for the rams, that can either advance or retreat, is manually controlled by the operator. The equation of state of olivine is used as an *in situ* pressure standard; sample length measurement from X-ray radiograph as a function of time is used as a strain indicator. Even though the speed of rams can be controlled, the strain rate of the samples may slightly fluctuate during each test. The incoherence between the speed of the ram and the flow rate of the sample are caused by both the manual operation and the physical properties of the pressure medium in the cell. The resulting constant strain rates can range from typically 5×10^{-6} to $5 \times 10^{-5} \text{ s}^{-1}$, with fluctuations no larger than typically a few 10^{-6} s^{-1} . The collected X-ray radiographs are analyzed after the experiment using an image analysis program (see *strain analysis* section) to trace the movement of the strain markers. Strain rates with precision of 10^{-6} s^{-1} are derived.

In run San-37 the powder starting materials were first pressurized to 3.5 GPa, heated to 1473 K. After narrowing of peak widths demonstrating the removal of initial deviatoric stress, it was then strained with constant strain rate ($\sim 10^{-5} \text{ s}^{-1}$) at 1473 K, 1273 K and 1073 K for about one hour at each temperature, then at 873 K for several hundred seconds. Samples were then quenched and pressurized up to 6.5 GPa. At 6.5 GPa, temperature was raised up to 1373 K first, then down to 1173 K before quench and depressurization. During run San-72, the sample was first compressed to 5 GPa, then heated for one hour at 1300 K, then quenched. The pressure was then further applied to the final condition of

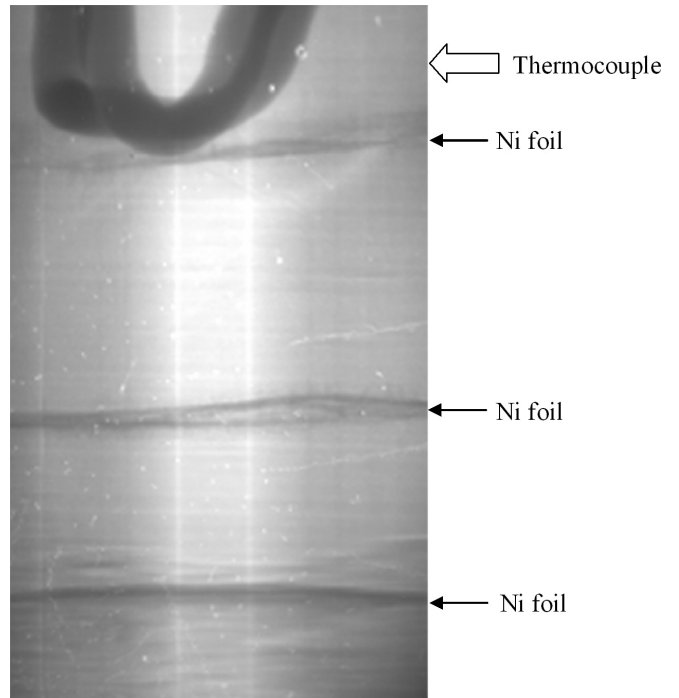


Fig. 2. Sample image at 6.5 GPa and 1273 K. The total height of the image is about 2 mm. The wires appearing on the top is the thermocouple. The three Ni foils serve as strain markers as well oxygen fugacity. The first layer of sample is *c* sample, the second layer of sample is *f* sample.

about 10 GPa, where it was heated to 1473 K, deformed at a constant strain rate, then cooled to 1273 K and further deformed, before final quench and depressurization. Sample Gar-25 was compressed to 5.2 GPa, heated to 1473 K and subjected to three speeds, two in the positive direction, and one negative. The results from only the positive directions are used here.

Data analysis

Pressure calibration

We use the olivine sample itself as the pressure standard using the 3rd order Birch-Murnaghan equation of state. The parameters used for the calculation (Isaak, 1992; Zha *et al.*, 1996) are listed in Table 1. The cell volume measured after the experiment is used as the reference $V_0 = 292.5769 \times 10^{-3} \text{ nm}^3$. Hydrostatic d-spacing (*a*, *b*, *c*) for orthorhombic olivine is derived by the following equation:

$$d(P, T) = (d_v + 2d_H)/3 \quad (1)$$

in which $d(P, T)$ is the hydrostatic cell parameters at *P* and *T*, d_v is the d spacing measured with vertical detector, d_H is the d spacing measured with horizontal detector. In this study, 3.5 (± 0.2) GPa and 6.5 (± 0.2) GPa are the two pressures during the experiment (run San-37), 9.6 (± 0.4) GPa at 1473 (± 50) K and 8.6 (± 0.4) GPa at 1273 (± 50) K for San-72 and 7.5 (± 0.4) GPa at 1473 (± 50) K for run Gar-25.

Table 1. Parameters in the EOS calculation

$K0$, GPa	128.8
$K0'$	4.2
$K0''$	0
$\alpha0(x10^5)$, K ⁻¹	3.4
$\alpha1(x10^8)$, K ⁻²	0.71
dK/dT , GPa K ⁻¹	-0.00156
$T(ref)$, K	298

Stress analysis

Differential stress is derived from the product of elastic moduli and lattice elastic strain. Lattice strain is manifested as the distortion of Debye ring with an orientation constrained by the geometry of the uniaxial deformation cell assembly. Lattice strain ϵ_{ij} and differential stress σ_{ij} are related by the following equation (3) (Weidner, 1998):

$$\epsilon_{ij} \sum_{k=1}^3 \sum_{l=1}^3 S_{ijkl} \delta_{kl} \quad (3)$$

$$\epsilon = [d(hkl) - d_p(hkl)] / d_p(hkl)$$

where $d(hkl)$ is the d spacing for each hkl , $d_p(hkl)$ is the spacing under hydrostatic pressure. The elastic compliance tensor S is derived from following equation (4) using experiment data from Isaak (1992) and Zha *et al.* (1996) (see Table 2), C is the elastic stiffness tensor, C_0 is the C at room pressure and temperature.

$$C = C_0 + T * dC / dT + P * dC / dP \quad (4)$$

$$S = C^{-1}$$

An analysis of the lattice strain for an orthorhombic system under non-hydrostatic pressure is described in detail elsewhere (Singh *et al.*, 1998), in which a uniform stress for each diffraction peak is assumed. In the current study, the following equation (5) is used to derive stress:

$$\sigma^{hkl} = \sigma_1^{hkl} - \sigma_3^{hkl} \quad (5)$$

where σ_1 and σ_3 , varying for each (hkl) , represent the principle stress detected by detectors which are placed at an azimuthal angle equal to half of diffraction angle (two theta) and 90°. In this stress analysis, which is slightly different from other studies (Singh *et al.*, 1998), we emphasize that different populations of grains, depending on their orientations, may support different values of stress. Then the sample stress (force per unit area) is an average over these populations. Variations of the calculated stress with diffraction peaks reflect the departure of the actual stress field from an average of the individual stresses.

Strain analysis

The total (macroscopic) strain of a sample between two consecutive X-ray images is derived by calculating the displacement of the strain markers in the second image, using

Table 2. Elastic constants for olivine

$C_0 =$	[323.7	66.4	71.6	0.0	0.0	0.0
	66.4	197.6	75.6	0.0	0.0	0.0
	71.6	75.6	235.1	0.0	0.0	0.0
	0.0	0.0	0.0	64.62	0.0	0.0
	0.0	0.0	0.0	0.0	78.05	0.0
	0.0	0.0	0.0	0.0	0.0	79.04]
$dC/dT =$	[-.0340	-.0105	-.0094	0.0	0.0	0.0
	-.0105	-.0285	-.0051	0.0	0.0	0.0
	-.0094	-.0051	-.0286	0.0	0.0	0.0
	0.0	0.0	0.0	-.0128	0.0	0.0
	0.0	0.0	0.0	0.0	-.0130	0.0
	0.0	0.0	0.0	0.0	0.0	-.0157]
$dC/dP =$	[7.98	4.74	4.48	0.0	0.0	0.0
	4.74	6.37	3.76	0.0	0.0	0.0
	4.48	3.76	6.38	0.0	0.0	0.0
	0.0	0.0	0.0	2.17	0.0	0.0
	0.0	0.0	0.0	0.0	1.64	0.0
	0.0	0.0	0.0	0.0	0.0	2.31]

the first image as a reference. The strain markers are Ni foils (each 25 μ m in thickness) in this study and are shown as the dark lines in the image. The principle of image analysis has been described in detail elsewhere (Li *et al.*, 2003). The precision is 10^{-4} for strain. In summary, the classic image-processing algorithm that yields sub-pixel resolution (Pratt, 1991; Trucco & Verri, 1998) was used. There are two steps in the image-analysis process. The first is the calculation of the sum of squared differences (SSD) between the two images using equation (6).

$$SSD_k = (\sum_i \sum_j [F_1(i,j,0) - F_2(i,j,k)]^2)^{1/2} \quad (6)$$

$$Coeff_k = SSD_k / \text{Max}[SSD_0, \dots, SSD_k] \quad (7)$$

In Equation (6), SSD_k is the root mean square of SSD of intensity between two images, $F_1(i, j, 0)$ and $F_2(i, j, k)$ indicate respectively the intensities of the first image and the second image with i as row and j as column, k is the shift position along the direction perpendicular to the Ni foil in the second image. The obtained SSD is then normalized as indicated by Equation (7), in which $Coeff_k$ is the normalized SSD_k . $Coeff_k$ as a function of k , can usually be plotted as a parabolic curve, which is in the minimum when the strain markers in two consecutive images match. The shift of k , then, quantifies the strain marker displacement, *i.e.*, the strain marker displacement is equal to k when $Coeff_k$ is the minimum among $Coeff_0$ to $Coeff_k$.

The second step is the parabola interpolation. It is to locate the extreme of a parabola interpolating values in the output of SSD as a function of displacement k . One hundred points were interpolated in between 2 pixels (using the cubic-spline interpolation method). This interpolation achieves a precision of 0.05 pixel, which results in a resolution better than 10^{-6} s⁻¹ on the measured strain rate between two images (for a 100-second time interval).

Creep results

Elastic stress *vs.* total (elastic + plastic) strain during deformation at different temperatures and pressures are illustrated in Fig. 3 and 4. The plotted stress, in Fig. 3 for the **f** sample and in Fig. 4 for the **c** sample of San-37, is the averaged value for four different diffraction peaks {(112), (130), (131), (021)}. The bar on each stress represents the span of stress for these peaks. Figure 5 shows the total strain as a function of time for both the **f** sample and the **c** sample. The **f** sample of San-37 experienced a total strain of about 30 %, while **c** sample experienced about ~17 % of total strain. At each temperature, a line was fitted for the strain data in a region where strain rate appears relatively constant; the strain rates are marked above/below the fitted lines. The variations in strain rates for given experimental conditions can be caused by several factors such as fluctuations of the driving system, the properties of the cell assembly, or the plastic properties of the sample such as dislocation-nucleation, time duration to reach steady state and/or work hardening.

At the highest explored temperatures (1473 K and 1373 K) the stress is quite constant throughout the entire deformation region. At 1273 K and 1173 K the stress increases initially and becomes constant at high strains, while at 1073 K the stress increases, but may not have reached steady state condition (constant flow stress) during the experiment. We interpret the stress increase at constant strain rate as work hardening, with the stress reaching a saturation, or steady state limit. At the highest temperatures, samples reach rapidly steady state (rapid equilibration of the defect microstructures), while at lower temperatures, steady state does not occur until after a few percent of strain. At the lowest explored temperature, steady state may not have been reached. We only discuss (in the discussion section) the results from the observations above 1273 K, where the data indicate that steady state was achieved. In general, in the San-37 experiment, the **f** sample supports higher stresses that are up to ~0.5 GPa higher than the **c** sample in the different temperature regions (*e.g.* 1173 K at 6.5 GPa, 1073 K at 3.5 GPa). For this experiment, we use an axial thermocouple placed only one side of the specimens, which broke the

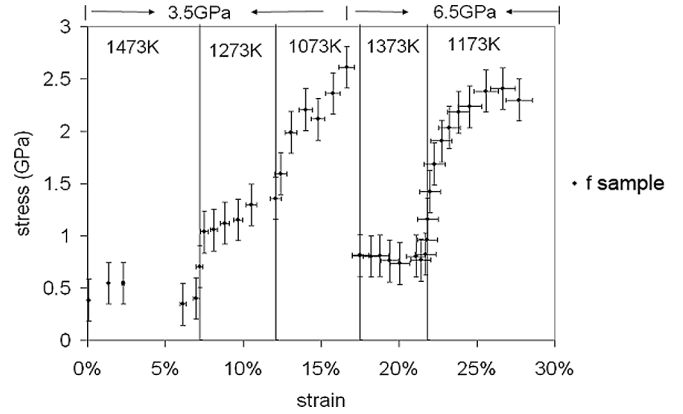


Fig. 3 Stress *vs.* strain for **f** sample of San-37. The strain represents the total of plastic and elastic strain as measured from X-ray imaging. The stress is averaged from X-ray diffraction data for peak [021], [130], [131] and [112] of **f** sample. Error bars represent the span of the four diffraction peaks. Horizontal error bars represent the error of the strain measurements.

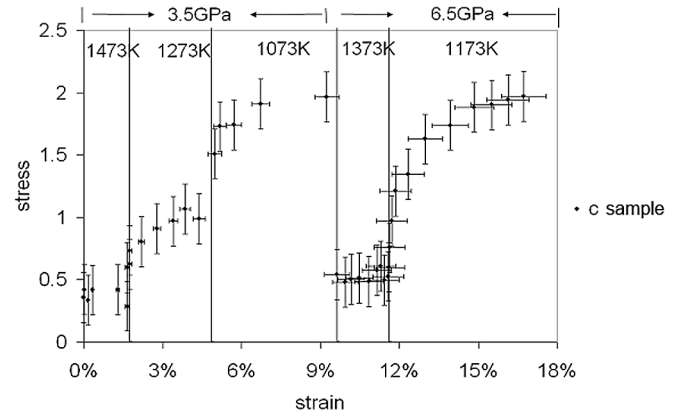


Fig. 4. Stress *vs.* strain for **c** sample of San-37. The strain represents the total of plastic and elastic strain as measured from X-ray imaging. The stress is averaged from X-ray diffraction data for peak [021], [130], [131] and [112] of **c** sample. Vertical bars represent the span of the four diffraction peaks. Horizontal error bars represent the error of the strain measurements.

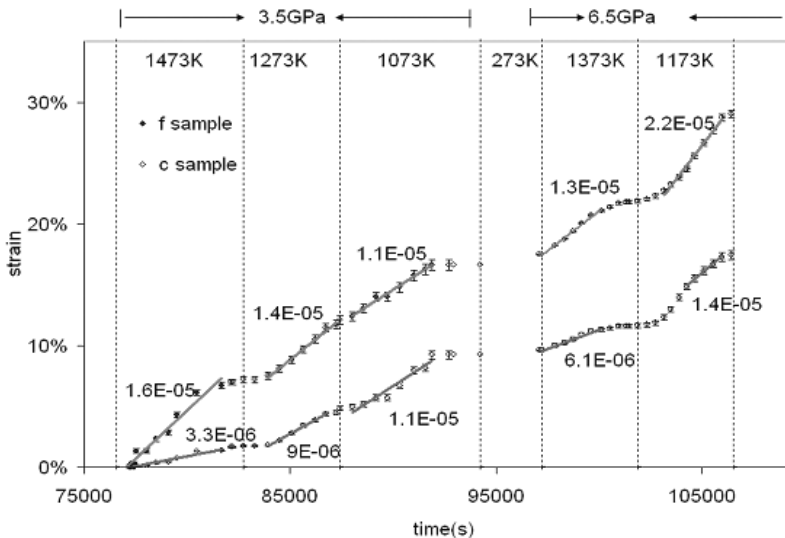


Fig. 5. Strain as a function of time for **f** and **c** samples of run San-32. Only the strains at steady state are fitted for strain rate. In general, the strain rates are in the scale of 10^{-5} s^{-1} , there are some fluctuations at each condition.

axial symmetry of the cell. A solid corundum piston was at one end of the specimen and a four-hole corundum piston was on the other end which was the passage way for the thermocouples. Even though the grain size of the two samples was very similar during the main part of the deformation, we observed different total strain and stresses in the two samples. The lower stress observed in the *c* sample is close to the four-hole piston end. We also found that the *f* and *c* samples experience different strain rates, with the slowest strain rate corresponding to the lowest stress. We interpret this difference as reflecting the different stress state in the cell resulting from the asymmetry of the cell design. As a consequence, the two samples yield different points on a stress – strain rate curve at the same pressure and temperature. Stresses and corresponding strain rates obtained for all specimens in steady state regimes (at constant flow stress) at different P-T conditions are reported in Table 3. These data are interpreted as representative of steady state flow properties of olivine at mantle pressures and temperatures.

The stress-strain relation for San-72 and Gar-25 are similar to those illustrated here for San-37. Total strains in excess of 20 % are achieved for both samples, with little strain hardening observed at 1473 K and similar behavior to San-37 at 1273 K.

Deformed specimen microstructures

Specimens for optical microscopy and TEM investigation were cut in optically selected areas of doubly polished petrographic sections (about 25 μm thick) prepared from specimens San-37 and Gar-25 slices cut near the center of the cell assembly. Both *f* and *c*, as well as samples from San-72 and Gar-25 exhibit comparable average grain size after the run, around 10 μm , with few grains reaching up to 80 μm in size, as observed by optical microscopy in-between crossed Nichols (examples shown in Fig. 6). This shows that grain growth was an active process during the run. Big grains of olivine

ten exhibit undulose extinctions in-between crossed nichols (Fig. 6), likely resulting from the presence of sub-grain boundaries, which suggests that dislocation climb was an active process at high temperature. TEM investigation of the deformed samples confirms these observations (below).

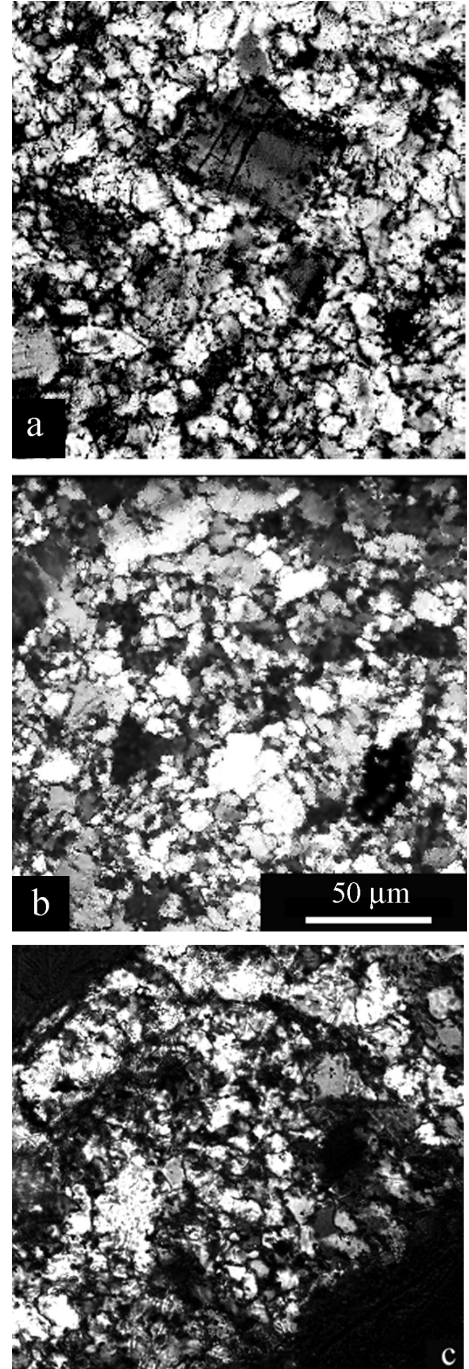


Fig. 6. Crossed-nichols transmitted-light black and white photomicrographs of sample petrographic sections (about 20 μm thick) cut from the middle of run products: a) fine-grain starting-material sample (San-37), b) coarse-grain starting-material sample (San-37), and c) Gar-25. Note that all samples exhibit similar grain size after the run, about 10 μm average with some grains reaching up to 80 μm in size. Note that some large grains (e.g. in center of figure a) exhibit undulose extinction.

Table 3. Stress and strain rate during steady state deformation.

	P, GPa	T ^a , K	σ , GPa	$\dot{\epsilon}^b$, s ⁻¹
San-37 <i>f</i>	3.5(2)	1473(50)	0.5(3)	1.60×10^{-5}
San-37 <i>f</i>	3.5(2)	1273(50)	1.3(3)	1.40×10^{-5}
San-37 <i>f</i>	3.5(2)	1073(50)	2.6(3)	1.10×10^{-5}
San-37 <i>c</i>	3.5(2)	1473(50)	0.4(3)	3.30×10^{-6}
San-37 <i>c</i>	3.5(2)	1273(50)	1.0(3)	9.00×10^{-6}
San-37 <i>c</i>	3.5(2)	1073(50)	2.0(3)	1.10×10^{-5}
San-37 <i>f</i>	6.5(2)	1373(50)	0.8(3)	1.30×10^{-5}
San-37 <i>f</i>	6.5(2)	1173(50)	2.4(3)	2.20×10^{-5}
San-37 <i>c</i>	6.5(2)	1373(50)	0.5(3)	6.00×10^{-6}
San-37 <i>c</i>	6.5(2)	1173(50)	2.0(3)	1.40×10^{-5}
San-72	9.6(4)	1473(50)	0.1(3)	5.0×10^{-5}
San-72	8.6(4)	1273(50)	1.0(3)	1.6×10^{-5}
Gar-25	7.5(4)	1473(50)	0.2(3)	3.0×10^{-5}
Gar-25	7.5(4)	1473(50)	0.1(3)	1.5×10^{-5}

The numbers in parentheses represent the uncertainty in the least significant digit.

^a W3 %Re-W25 %Re thermocouple. T is calibrated based on Power-T at 6.5 GPa.

^b Estimated error for strain rate is $\sim 0.5\%$.

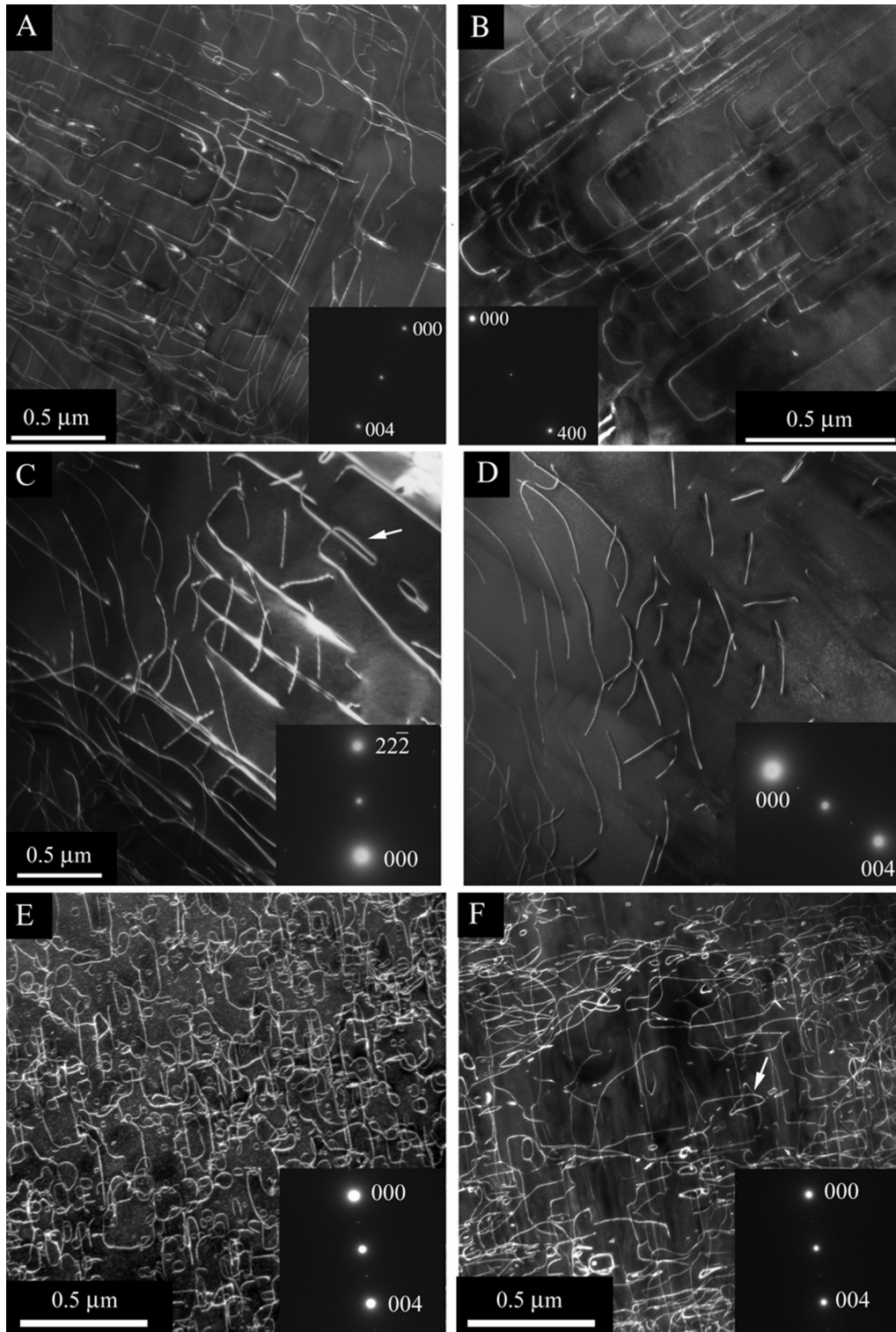


Fig. 7. Dark-field weak-beam TEM micrographs of the deformed samples after run San-37 and corresponding diffraction patterns (insets). a) and b), respectively **c** and **a** dislocations well confined in their (010) glide plane, as observed in the f sample. c) and d), two micrographs of the same area in the **c** sample obtained with diffraction vectors $g = 222$ and $g = 004$, respectively; with $g = 222$, both **a** and **c** dislocations are visible, while **a** dislocations are out of contrast with $g = 004$. Note the evidence of **a** dislocation cross-slip (arrow) as an active mechanism during the run, and the weak interaction between dislocations having different Burgers vectors. e) Olivine grain exhibiting numerous sessile **c**-dislocations loops (out of their glide plane). These loops must have formed by cross-slip of screw dislocation segments (see text for details). f) Olivine grain exhibiting high density of mobile **c** dislocations (reaching $3 \times 10^{14} \text{ m}^{-2}$ in some area). Note that the screw dislocation segments are fairly straight while the more mobile edge dislocation segments tend to form tangle-like features. Note also the numerous evidence of **c**-dislocation cross-slip (*e.g.*, arrow), as well as the numerous tiny sessile dislocation loops visible throughout the image.

Sample petrographic sections were ion-thinned (with a 5-kV argon beam) to electron transparency. A Philips CM30 and a Jeol 200CX electron microscope, operating respectively at 300 and 200 kV, were used to investigate the specimens. The TEM microstructures observed in the deformed **f** and **c** samples are very similar. Because of this similarity, no distinction is made between samples in the following description. The dislocation microstructures, shown in Fig. 7, exhibit superimposed features characteristic of different deformation regimes. This is not surprising since the run P-T- σ cycle ended at moderate temperature (1173 K) and high stresses, while T reached 1473 K (high temperature with low stresses) during the run. Consequently, mobile dislocations appear fairly well confined in their glide planes (*e.g.*, Fig. 7a and 7b) in the quenched samples, although evidence of climb assisted dislocation glide such as sub-grain boundary fragments, which probably formed at the highest run temperature, are still observable in some area of the run products. Dislocations with both **a** and **c** Burgers vectors are observed (Fig. 7) with few interactions between dislocations with different Burgers vectors (Fig. 7c and 7d). The **c** dislocation slip system, however, largely dominated the deformation, *i.e.* **c** dislocations are present in all observed olivine grains and exhibit in some grains very high dislocation densities (up to about $3 \times 10^{14} \text{ m}^{-2}$ or higher, *e.g.* Fig. 7e and 7f). This observation suggests that, unlike **a** dislocation slip systems, **c** slip was very active at all temperature during the high-pressure run. This result, expected below typically 1100 K in a high-stress deformation regime, is unexpected at higher temperature and lower stress level where **a** slip systems dominate polycrystalline olivine deformation in 1-atm experiments. It is however consistent with results of recent high pressure deformation experiments reported by Couvy *et al.* (2004), suggesting that the **c** slip may be dominant at 11 GPa and 1673 K. The **c** slip has also been shown to be dominant at high temperature in wet olivine (Jung & Karato, 2001).

Evidence of **a** and **c** screw-dislocation cross-slip is also observed (*e.g.* Fig. 7c and 7e or 7f, respectively). This mechanism, however, seems to be occasional in the case of **a** dislocations, while it appears to strongly contribute to **c** dislocation mobility at high pressure (Fig. 7e and 7f). Figure 7e shows an area of the **f** sample exhibiting numerous sessile **c**-dislocation loops (out of the glide plane). Such sessile loops can form at high temperature from equilibrated dislocation dipoles that evolve toward dislocation loops by diffusion. However, given the relatively high stress applied on the specimen during the experiment, it is unlikely that such dipoles were stable during the run. We thus conclude that the observed sessile loops formed by cross-slip of **c** screw dislocations. More direct evidence of **c**-dislocation cross-slip is also visible throughout the samples (*e.g.* Fig. 7f). Cross-slip of **c**-dislocations has been previously observed by Phakey *et al.* (1972) in experimentally deformed Fo_{92} forsterite under moderate (1 GPa) confining pressure. This suggests that the **c** dislocation cross-slip mechanism may be particularly active at high pressure. Although it strongly depends on the orientation of each individual olivine grain with respect to the applied stress, cross-slip may assist the motion of **c**-dislocation in olivine at high

pressure. This may contribute to the apparent high mobility of **c**-dislocations in our samples with respect to that of **a**-dislocations.

Discussion

Here, we present deformation data on olivine obtained in a multi-anvil high-pressure device and compare these results with lower pressure data. These experiments take full advantage of the synchrotron X-ray measure of stress and strain as well as the unique deformation features of the D-DIA. The data reported here do not have enough range in temperature and strain rate to derive a well-constrained rheological flow law for olivine. However, they do span a wide pressure range at relatively constant water content. We, therefore, examine these data to constrain the pressure dependence of olivine flow and define the degree of consistency with the existing data.

We follow the analysis used by Karato & Jung (2003) and map the observed data that are taken at different temperatures and strain rates to define the flow stress at a common strain rate and temperature using an activation energy $E^* = 470 \text{ kJ/mol}$ for temperature correction and a stress exponent $n = 3$ (Mei & Kohlstedt, 2000b) for stress correction, using a flow law of the type:

$$\dot{\epsilon}(T, P) = A C_{OH} \sigma^n \exp\left(-\frac{E^* + PV^*}{RT}\right), \quad (8)$$

where $\dot{\epsilon}$ is the strain rate, A is a constant, C_{OH} is the OH-defect concentration in olivine, V^* is the activation volume, and R is the gas constant. The activation volume V^* , that expresses the effect of pressure on olivine flow, has not been well constrained in the past, mostly because of limitation in the pressure range accessible in deformation experiments. Some studies (*e.g.* Kohlstedt & Wang, 2001; Karato & Wu, 1993; Kohlstedt *et al.*, 1980; Ross *et al.*, 1979; Borch & Green, 1987; Karato & Jung, 2003) give V^* values varying from 11–27 cm^3/mol .

Figure 8 illustrates the flow stress that is deduced from correcting the observations to 1473 K and a strain rate of 10^{-4} s^{-1} but with no explicit correction for water concentration, C_{OH} , since water content is constant in all of our samples except the ‘dry’ sample, Gar-25. For this figure, we use only the data we obtained at temperatures of 1273 K or greater, as these are the likely candidates for describing power-law-creep deformation. The lower temperature data are probably dominated by dislocation-glide type process with limited dislocation climb; they are thus not representative of mantle deformation and are not further discussed in this article. In Fig. 8, the data from both the **c** and **f** samples of run San-37, and samples San-72 and Gar-25, are plotted as a function of $P \cdot (1473/T_{ob})$, where T_{ob} is the observed temperature. The slope of a flow law on this semi-log plot is proportional to $V^*/(R \cdot 1473)$. Since C_{OH} was constant in our olivine samples throughout the deformation experiments for San-37 and San-72 (see ‘water content’ sub-section), these samples in Fig. 8 show the effect of pressure alone on olivine plastic flow. Focusing on the data reported here (solid symbols), the error bars indicate the range of stress compati-

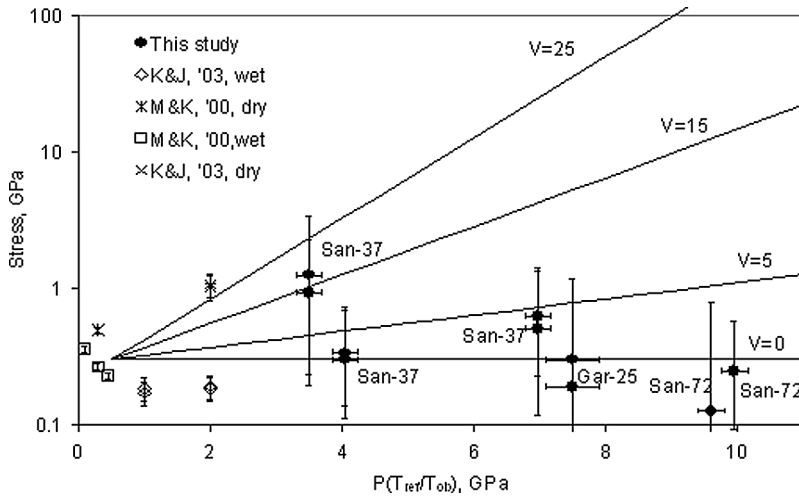


Fig. 8. Flow stress for olivine samples corrected to a reference temperature of 1473 K and a strain rate of 10^{-4} s^{-1} . Equation (8) is applied to correct the measured stress at the measured strain rate and temperature to these conditions, using $n=3$ and $E^*=470 \text{ kJ/mol}$. Water content is assumed to be constant and pressure is corrected to $P \cdot (1473/T_{ob})$ to enable straight lines to represent a constant activation volume. The solid symbols are for this study runs San-37, San-72, and Gar-25. The error bars reflect the uncertainties of Table 3. The closed open symbols are for wet samples while the cross and plus symbols are for dry samples. M&K, '00 is data of Mei & Kohlstedt (2000b), K&J, '03 is the data of Karato & Jung (2003). The solid lines illustrate the slopes of flow laws corresponding to several activation volumes. The numbers are in units of cm^3/mol . We conclude that an activation volume of $0 \pm 5 \text{ cm}^3/\text{mol}$ fits the data.

ble with the errors quoted in Table 3. A change of activation energy by 100 kJ/mol does not appreciably alter the character of the figure.

The extrapolated values for the **c** and **f** samples of San-37, as reported in Fig. 8, differ by no more than 30 %. If an exponent, n , of 1 is used, then the flow stresses from the two different samples differ by as much as 400 %, outside of the errors of the individual measurements. A value of n of 3.5 yields the best agreement between the two samples with a misfit of no more than 20 %. From this we conclude that data from San-37 are not consistent with diffusion creep, and can only be fit with power-law creep flow, consistent with the TEM observations indicating the activity of dislocations. This gives us confidence in the choice of parameter, $n=3$, used in this analysis.

The best fit to our data alone yields an activation volume, V^* , of $-5 \pm 10 \text{ cm}^3/\text{mol}$. A negative activation volume, while not required by the data, is not what is usually expected. Mei & Kohlstedt (2000a, b) indicate that silicon diffusion, controlled by silicon interstitial defects, may be the rate limiting process (through dislocation climb) for plastic deformation in water bearing olivine. If this is the case, then one expects that the activation volume for creep will be related to that for silicon self-diffusion in olivine, as described by Bejina *et al.* (1997 and 1999), who report a slightly negative activation volume for this latter process.

Regarding the effect of water on olivine plasticity at high pressure, the flow stress for Gar-25 is indistinguishable from the other data even though the water content of Gar-25 was about an order of magnitude smaller than that of the other samples. We thus conclude that, within our experimental error, the water content of the sample at these concentrations of water, does not significantly affect the flow properties of olivine.

Figure 8 also compares our data with previously reported data obtained at significantly lower pressure. As representative of wet olivine, we refer to the recent study by Mei & Kohlstedt (2000b). The flow stress data of Mei & Kohlstedt (2000b) are for saturated olivine in the power-law creep regime. The stress decreases with increasing pressure as the water concentration increases. The data were obtained with

a gas-pressure device equipped with high precision stress measuring capabilities. These data clearly indicate a weakening effect of water on the flow strength of olivine. Their highest pressure data has less water incorporated in the olivine structure than is present in our polycrystals, even including our 'dry' Gar-25 sample. However, if we compare our water content with the water content that they obtain from their polycrystalline IR measurement, then our water content is similar to that of their 100 MPa samples and less than that of their higher pressure samples. All of their measured flow stress values fall within our experimental error and are thus quite consistent with our data as they represent the lower bound of the low pressure flow strength expected from an olivine sample with the observed water content as those reported here.

Also illustrated in Fig. 8 are data from Karato & Jung (2003). They add new data at three conditions to the existing olivine data, one at 1 GPa for dry olivine, and two for wet olivine, one at 1 GPa and the other at 2 GPa. Lacking a direct stress measure, they use dislocation density to define the stress of these samples. We illustrate their two wet olivine measurements in Fig. 8. Their observation is very similar to ours: namely stress is independent of pressure at constant strain rate. Since their samples were in a water present environment, they concluded that the water concentration in the olivine increased with pressure as demonstrated by Kohlstedt *et al.* (1996) at lower pressure and that this increased concentration of water caused the flow strength to decrease as reported by Mei & Kohlstedt (2000b). Their observed constancy of flow stress with pressure was thus interpreted as due to a strong increase in strength with pressure to compensate the decrease in strength due to the increase concentration of water. In our case, the water content did not increase with pressure. However, we cannot rule out the possibility that the partitioning of the water between grain boundaries and grain interior does not change with pressure. Thus, the constancy of the strength with pressure requires a small pressure effect on olivine flow (small activation volume V^*). This observation applied to the data of Karato & Jung (2003) then implies that the large concentrations of water in their samples no longer lowers sample strength. This inter-

pretation is consistent with our observation of the lack of effect of water content when comparing Gar-25 plasticity with that of our other samples. Thus, the constancy of strength found by Karato & Jung (2003) can be interpreted as due to a small dependence of olivine strength on either water content or pressure, a conclusion in good agreement with our observations.

Also plotted in Fig. 8 are dry olivine flow stress data from Mei & Kohlstedt (2000b) and Karato & Jung (2003). These data represent an upper bound to the low pressure flow strength of olivine samples with a water content of the samples reported here. The upper bound defined by the dry olivine flow strength and the lower bound defined by the wet flow strength of these studies are quite consistent with the flow strength reported here, but provide a more accurate low pressure value than is possible with the data reported here. These data thus assist in narrowing the possible range for the activation volume of the olivine samples studied here.

Karato & Jung (2003) concluded a strong pressure effect on the flow strength of dry olivine. This conclusion was mostly based on the inferred strength of about 100 GPa of dry olivine at 15 GPa confining pressure from Karato & Rubie (1997). The study of Karato & Rubie (1997) did not measure stress, but only estimated it using several assumptions about the sample assembly, the friction of the pistons in a multi-anvil system, and the relative strengths of the sample and piston. Their experiment was a relaxation experiment, based on recovery examination of the specimen. In fact, the strength of olivine they report is about an order of magnitude greater than the measured strength of diamond at comparable conditions (Weidner *et al.*, 1994b). Such a high strength of the sample will violate some of the assumptions made to calculate the stress. Thus, we have not included their data here.

The comparison of all of these data need to recognize that free water was present in the experiments by Mei & Kohlstedt (2000b) and Karato & Jung (2003) but not in the current experiments. As a result, the water content in our samples, while fixed, is less than the highest water content of Karato & Jung (2003) and the water actually incorporated in the olivine lattice may be less than all but the dry samples illustrated here. Thus, if water weakens olivine, our back-extrapolated low pressure flow stress should be equal to or higher than the saturated data. With all of this data we define a maximum pressure effect. We conclude that the activation volume V^* for high-temperature olivine dislocation creep, is $V^* = 0 \pm 5 \text{ cm}^3/\text{mol}$. This value is consistent with our data and consistent with the constraints imposed by the low pressure observations of previous studies, as illustrated in Fig. 8.

Such a small activation volume for olivine dislocation creep, although previously suggested (*e.g.*, Béjina *et al.*, 1999), does not agree with the larger activation volumes up to $27 \text{ cm}^3/\text{mol}$ (*e.g.* Green & Borch, 1987) found in the literature. Beside the studies of Mei & Kohlstedt (2000b) and Karato & Jung (2003) already discussed above, the early works of Ross *et al.* (1979) and Green & Borch (1987) (see also Borch & Green, 1987) were carried out at relatively low pressure (less than 2.5 GPa) in Griggs-type solid-medium devices. Green & Borch (1987) flow stress at 2.5 GPa (300 MPa at $2 \cdot 10^{-4} \text{ s}^{-1}$ and 1473 K) agrees with the flow stress measured in this study. Their flow stress at lower pres-

ures, however, appears to be low relative to further studies such as Mei & Kohlstedt (2000b). Ross *et al.* (1979) report values of V^* ranging from 10.6 to $15.4 \text{ cm}^3/\text{mol}$, and Green & Borch (1987) report $V^* = 27 \text{ cm}^3/\text{mol}$. The Griggs-type apparatus, plagued by poorly constrained stress and strain rates, coupled with the relatively low pressure range, cannot provide a well defined activation volume that is appropriate to mantle conditions. Furthermore, the activation volumes that have been reported may reflect processes that are operative at low pressure such as grain-boundary sliding accommodating dislocation creep, but that become inactive at high pressure. Indeed, grain-boundary migration (through dynamic recrystallization) was implicated as assisting olivine dislocation creep at mantle conditions (*e.g.*, Li *et al.*, 2003). A change in the grain-boundary processes assisting dislocation creep is likely to induce a change in the apparent dislocation-creep activation volume, which could explain the apparent decrease of V^* with pressure.

Due to experimental limitations, very few studies, other than those already discussed, explored the olivine dislocation-creep V^* at $P > 3 \text{ GPa}$. However, there are some studies that have been carried out at similar pressures to those reported here on related phenomena that do provide insights into the activation volume for creep. Deformation experiments have been carried out on olivine single-crystals in a multi-anvil press at pressure and temperature up to 13.5 GPa and 1873 K by Bussod *et al.* (1993), who report that olivine crystals, after a fracturation and cataclastic deformation episode during cold compression, dynamically recrystallized during deformation at mantle conditions, while dislocation creep was an active process. Except for the measure of strain, deduced from the deformation of the apparatus during the runs, these experiments were mostly interpreted *post-mortem* from the microstructures observed in the run products. Bussod *et al.* (1993) report an activation volume V^* ranging from 5 to $10 \text{ cm}^3/\text{mol}$ for dislocation creep assisted by dynamic recrystallization, a value significantly lower than those reported from low pressure experiments and in good agreement (within uncertainties) with our observations. Karato *et al.* (1993) investigated the activation volume for dislocation recovery (dislocation climb) in olivine at pressure and temperature up to 10 GPa and 1773 K, respectively. They report an activation volume $V^* \sim 6 \text{ cm}^3/\text{mol}$ for this process which mostly involves ionic/vacancy bulk diffusion. Although the reported V^* can characterize the Navarro-Herring deformation process (*i.e.* the bulk-diffusion creep), it can also characterize the dislocation-climb controlled dislocation-creep process which, according to most authors, dominate olivine high-temperature deformation. Thus, Karato *et al.* (1993)'s result suggests that the activation volume for olivine dislocation creep at mantle conditions is low; a result in reasonable agreement with our observations. Béjina *et al.* (1999) also report a low activation volume $V^* \sim 5 \text{ cm}^3/\text{mol}$ for olivine dislocation creep at mantle conditions. This value was deduced from Mg and Si self-diffusion coefficients in olivine (see also Béjina *et al.*, 1997) as measured at pressure and temperature up to 9 GPa and 1873 K, respectively. To link the diffusion data to dislocation creep, they assumed a dislocation-climb controlled dislocation-creep deformation process in the frame-

work of the Jaoul (1990)'s multicomponent diffusion model. Béjina *et al.* (1999) conclusion is also in good agreement with our present observation of a low activation volume for olivine dislocation creep at mantle conditions.

Conclusion

This is the first paper presenting olivine flow properties using a D-DIA at a synchrotron. We demonstrate that flow reaches steady state at most conditions of our study. This occurs for total strains of a few per cent.

Our TEM observations on the recovered samples suggest that **a** and **c** dislocation glide were active during sample deformation, and assisted by dislocation climb (at high temperature) as well as dislocation cross slip. Dynamic recrystallization, as observed from previous studies (Li *et al.*, 2003), is consistent with the evolution of grain size observed here. The absence of grain size dependence of flow in the previous studies of Li *et al.* (2003) along with the value of *n* and the TEM observations suggests that diffusion creep is not a dominant process in these experiments. We infer that the high-temperature flow is in the 'power-law' creep regime.

We find the steady state rheological data measured at high pressure (> 3 GPa) are similar to the reported data at room pressure or low pressure (< 3 GPa). Our data do not span sufficient parameter space to derive a well constrained flow law, however, the good agreement with reported room/low pressure flow laws suggests that flow parameters such as activation energy, stress exponent, and pre-exponential terms are quite consistent with our data. Taken together with the low pressure data on both wet and dry olivine, we conclude that the activation volume for high-temperature olivine dislocation-creep is $0 \pm 5 \text{ cm}^3/\text{mol}$.

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